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(72) Inventors HENRI MANGIN and MICHEL HUCHETTE

(54) PROCESS FOR PRODUCING CRYSTALLINE SORBITOL

(71) We, ROQUETTE FRERES SOCIETE ANONYME, of 62 - Lestrem (France), a French company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to a process for the continuous manufacture of crystalline sorbitol.

It is already known how to manufacture crystallized sorbitol by adding to concentrated sorbitol solutions fine sorbitol particles which serve as crystallization agents, the aggregate being violently stirred; a white mass is thus obtained which crystallizes at the end of a duration varying from several hours to several days so that a solid mass which is pulverized and sieved is obtained.

This process presents the disadvantage of being discontinuous, of absorbing a lot of power, of often being manual, of causing many difficulties in sticking and handling and of giving a quality of sorbitol which does not meet the required stability standards.

It has also been suggested that crystalline sorbitol could be obtained by adding a concentrated solution of sorbitol to sorbitol powder but this leads to a deposit of crystallized sorbitol on the walls of the pulverization chamber.

This process presents the disadvantage of requiring very frequent stopping and cleaning of the apparatus, of recycling large amounts of powder; this process gives an unstable quality of sorbitol and is not completely continuous.

It was finally suggested that the sorbitol was crystallized starting with concentrated solutions having 70 to 98% of dry matter and using as solvents alcohols such as methanol, the crystallized mass obtained being drained and then dried.

This process presents the disadvantage of entailing the handling of a solvent which later has to be completely eliminated from the product; this process is moreover expensive, gives a very low yield and provides a quality of sorbitol which is difficult to control from the point of view of particle size and stability.

The invention has as its particular aim the providing of a process for manufacturing crystalline sorbitol which is less susceptible to aforementioned disadvantages and which enables crystalline sorbitol to be obtained in a stable form at a relatively low cost price.

According to the present invention, there is provided a process for the manufacture of crystalline particles of sorbitol, which comprises continuously mixing molten sorbitol having a dry matter content of at least 90% by weight with from 20 to 80% by weight of powdered sorbitol based on the total weight of molten and powdered sorbitol, the mixing being effected by simultaneously dispersing the molten and powdered sorbitol into an open rotating receptacle containing granules of conglomerated molten sorbitol and sorbitol powder whereby the molten and powdered sorbitol are mixed at the surface of the sorbitol contained in the receptacle, collecting sorbitol granules from the receptacle and crystallising molten sorbitol contained in said granules, the sorbitol in the receptacle being maintained in motion by the rotation of the receptacle and being maintained at a temperature of at least 90°C.

In carrying out the process of the invention, the molten sorbitol is preferably introduced into the receptacle in a sub-divided form, for example in the form of droplets or globules, jets or bundles of jets or sheets or layers of sheets..

According to a preferred method of carrying out the abovementioned process, some molten sorbitol, with a content in dry matter of over 98%, is brought to a temperature greater than 95°C, most preferably about

100°C., and continuously mixed with a substantially equal amount of sorbitol powder having a particle size lower than 5mm inside an open rotating receptacle, on the surface of the moving mixture, the dimensions, the inclination, if any, and the speed of rotation of the receptacle being chosen such that the product collected from the receptacle appears in the form of granules with a diameter of 1mm to 50mm.

The process of the invention may be carried out in apparatus comprising an open rotating receptacle, with a rotation axis possibly horizontally inclined, means for bringing to an area situated inside the receptacle, above the mass which partially fills it, and dispersing therein some molten sorbitol, preferably sub-divided into the forms referred to above and some sorbitol in powder form preferably having a particle size of less than 5mm and means to ensure the mixing of the molten sorbitol and the powdered sorbitol on the surface of the moving mass partially filling the receptacle.

According to an advantageous method of carrying out the invention, the aforesaid plant comprises, in series:

— an evaporation chamber under vacuum suitable for bringing a solution of sorbitol to a content in dry matter of over 98%,

— preferably a chamber for stocking the practically anhydrous molten sorbitol thus obtained, inside which it is kept at a temperature over 95°C, preferably in the neighbourhood of 100°C.,

— means for the dispersion of the anhydrous molten sorbitol at a temperature of at least 95°C, in the receptacle and

— means arranged to ensure the mixing of the dispersed molten sorbitol and the sorbitol powder inside the receptacle.

The sorbitol particles collected from the receptacle may be matured to increase their crystallinity by transferring the granules to a rotating cylinder with dimension characteristics and rotation characteristics such that the duration of stay of the granules coming from the receptacle is sufficient to ensure crystallization of the sorbitol. The granules may then be passed to means for grinding and sifting.

The process of the invention will now be described in more detail with particular reference to the accompanying drawings, of which

Figure 1 shows diagrammatically the various constituent parts of a plant suitable for carrying out the process of invention.

Figures 2 and 3 show in diagrammatic section on another scale a constituent part of the aforementioned plant arranged according to the two variants.

In carrying out the process of the invention in the plant illustrated, a solution of sorbitol, of the type available in the trade, that is to say which may contain other polyalcohols such as mannitol, and the content of dry matter of which is of the order of 70% is first dehydrated up to at least 90% of dry matter, and preferably up to at least 98% of dry matter, then dispersed at a temperature of over 95°C, usually in the neighbourhood of 100°C, in the form of globules inside an open rotating receptacle 1, the axis of rotation XY of which may be inclined on a horizontal plane H at an angle α. Some solid sorbitol in the form of powder is also introduced into the receptacle and is mixed with the dispersed molten sorbitol, the mixture forming on the surface of the mass of sorbitol which partially fills the receptacle and which comprises a mixture of the molten and powdered sorbitol added previously.

Instead of dispersing the molten sorbitol in the form of globules, it can also be dispersed inside the receptacle in the form of bundles of jets, sheets, or layers of sheets or otherwise.

As regards the sorbitol powder going into the aforesaid mixture and the constituent particles of which serve as crystal nuclei, a particle size of less than 5mm and most preferably of the order of 0.5mm is preferred.

In order to prepare the molten mass of sorbitol with a content of dry matter of over 90%, the aforementioned solution of sorbitol is brought under a vacuum of several millimetres of mercury, usually less than 20 mm of mercury, at a temperature of the order of 130 to 140°C and this temperature is maintained whilst stirring for a sufficient time to reach a degree of dehydration down to about 0.5% in water. It is noted moreover that duringf the dehydration stage, the temperature first rises rapidly up to about 80°C, keeping at this temperature for a certain period until the greater part of the water contained is removed — usually the stage in question lasts 1 to 2 hours — then it rises rapidly to 130 to 140°C during which time the water content drops to about 0.5%.

The proportions in the mixture made up of molten sorbitol and sorbitol in powder form are such that there is at least 20% of powdered sorbitol; in practice, practically equal ponderal proportions are used.

The mixing is effected on the surface of the mass in motion filling the receptacle partially; the motion in question brings to mind that of a mass of pills inside a pill making machine and it is found that granules which are bigger and bigger are formed, the largest granules having the tendency to came to the surface of the mass in movement.

The temperature of the mass in motion is kept at a level of over 90°C and usually

lies between 90 and 95°C, as a result of which the particles of sorbitol are obtained which are matured to increase their crystallinity so as to provide a stable form which melts at a temperature of 95 to 98°C and which complies with the standards of pharmacopoeia (It should be mentioned in this connection that the form called "non stable" melts at temperature of less than 90°C and and if 'isomerised" to a stable form, the sorbitol particles stick to one another.).

It is in particular large size granules which are collected by overflow at the outlet of the receptacle.

The size of the granules is conditioned by the characteristics of the abovementioned receptacle.

The granules consist of a conglomerate of molten sorbitol and crystalline powder of sorbitol which it is fitting to cause to "mature" in order to make the molten sorbitol crystallize.

This stage of maturing can be achieved by keeping the granules moving at a temperature of 50 to 95°C, preferably of 80 to 90°C, during approximately 1 to 20, but preferably 4 to 6 hours, preferably in a current of air.

The crystallized particles of sorbitol may then be ground to the required particle size then sorted by sifting; the powder eliminated by sifting may then advantageously be recycled to the abovementioned receptacle for use as crystallization nuclei.

It should be noted that it is useful to carry out the abovementioned operations under conditioned air the characteristics of which can be the following:

relative humidity: 10 to 50%, preferably 15%;

temperature: 10 to 35°C, preferably 25°C higher than that of the granules.

Given this, the plant according to the invention for operating the process which has been described essentially comprises the aforementioned receptacle with the axis XY possibly inclined to the horizontal plane, the said receptacle being placed in series, as shown in Figure 1, with:

— an evaporation vessel under vacuum 2

— a tank or vessel for stocking of the molten sorbitol 3

— means for dispersion of the molten sorbitol inside the receptacle 1 and means suitable for mixing the molten dispersed sorbitol with the crystallized sorbitol in powder form and below the receptacle:

— a rotating cylinder 4 for "maturing" working like a turning kiln of the type used in metallurgy

— a pulverising plane 5,

— a sifting plant 6,

— means for recycling 7 at the outlet of the plant for sifting and means for returning part of the ground crystallised sorbitol to mixing means working within the receptacle 1.

The evaporation vessel 2 is fitted with a set of heating tubes 8 capable of bringing the solution of sorbitol up to 140°C, with a stirring system 9, a solution of sorbitol delivery pipe 10 and a vacuum inlet 12.

This vessel 2 is connected to the storage tank 3 by piping 13 fitted with a pump 13a.

The storage tank or buffer 3 is fitted with a tube bundle 14 suitable for maintaining the temperature of the molten sorbitol, stored inside, at a temperature in the order of 100°C. This tank is also equipped to its advantage with a stirring system marked 15.

It is the mass of molten sorbitol contained in the tank which allows the continuous feed of receptacle 1.

This storage tank could however be eliminated and the receptacle 1 alternatively fed from two tanks of the type 2 successively and alternatively connected to the receptacle 1 when the sorbitol which they contain is dehydrated and this would also permit continuous working.

The means for dispersion of the molten sorbitol comprise a pump 16 taking the molten sorbitol in the tank 3 and dispersing it with the help of a nozzle 17, for instance tapered, placed in the neighbourhood of the bottom 1a of the receptacle. The globules of the dispersed molten sorbitol are mixed with the crystallised powder sorbitol which is introduced by means of a device 19 of the vibrating distributor type.

The mixture is made on the surface of the mass M in motion (molten sorbitol + crystallised sorbitol powder) which fills the receptacle as shown.

The rotation of this latter is ensured for instance by a drive mechanism diagrammatically shown at 20.

The receptacle 1 can be made in the shape of a tank or drum as shown in Figure 1.

It can also be made in a manner similar to a dredger, that is to say in the shape of a sphere from which the dome has been removed (see Figure 2).

It is also possible to make it as shown in Figure 3 in a manner similar to that shown in Figure 1 but comprising a circular edge 1b parallel to the bottom and turned inwards.

When the receptacle 1 has a shape like that represented in Figure 1, it is used with an angle of inclination α usually over 25° and, preferably lying between 25 and 45°.

Means not shown are provided in order to vary the said inclination α of the axis of rotation XY on the horizontal plane.

When the receptacle 1 is in a form similar

to those shown in Figures 2 and 3, the inclination can be lower than 25° and even become none with the receptacle of the type of Fig. 3.

The receptacle 1 is advantageously fitted with a scraper knife 21 (Figure 1).

The granules which are brought to the surface of the mass in motion M are cleared from the receptacle 1 by an overflow system (see arrow F_1) and thus carried to a chute 22 which is connected by piping 23 to the inlet of the rotating cylinder 4. This rotating cylinder 4 is supported by two pedestal bearings 24a and 24b which comprise the means for putting it in rotation. The dimensions of this cylinder, its speed of rotation and its inclination are chosen in such a way that the length of time a given granule remains inside this cylinder should preferably be between 4 and 6 hours.

At the outlet of the rotating cylinder 4, the matured granules, consisting of crystallised sorbitol, fall inside a chute 25 and are introduced into the aforementioned grinding plant 5. This plant is preferably arranged so that the particle size of the ground product obtained can be varied.

At the outlet of this grinding plant, the ground product is brought by piping 26, preferably fitted with pneumatic means of routing, to the sifting plant 6 at the outlet of which is collected on the one hand the product with the desired particle size which is cleared by piping 27, and on the other hand, a certain amount of product with too low a particle size which is recycled by piping 28 to the receptacle 1, the said piping 28 comprising the aforementioned means 7 which consist for instance of a turbine.

This being so and in order to clarify our views, we would point out that in a plant giving good results and one which has been tested by the applicant company, a receptacle 1 was used in the shape of a tank of the type shown in Figure 1, with a diameter of 2.60 m and a depth of 1.20 m and with an inclination which was adjustable between 25 and 45°. Generally this tank was given a rotation speed of approximately 7 revolutions per minute. In this same plant a cylinder 4 was used, the length of which is 8.50 m, the diameter 1.80 m, the inclination 5° and the speed of rotation 10 rev/min. The length of time that a given granule stays inside this cylinder 4 is in the order of 5 hours.

The following Examples illustrate the preparation of crystalline sorbitol by the use of the process according to the invention.

EXAMPLE 1

A solution of sorbitol of high purity the content in dry matter of which is about 70% is brought by piping 10 into the evaporation vessel 2. Once the required amount of sorbitol solution has been introduced into the vessel, the temperature is progressively raised to 125° C, the pressure being lowered to a value below 20 mm of mercury. Thus at the end of 2 hours molten sorbitol with a content in dry matter of 99.8% is obtained.

This molten sorbitol is put into the storage tank or buffer 3 from which it is continuously taken by means of the pump 16, which ensures its dispersion in the form of globules with a diameter of less than 0.1 mm by means of a nozzle 17 the diameter of which is 0.4 mm. The pressure under which the molten sorbitol is routed to the nozzle 17 is 3.5 kg/cm².

Simultaneously with this dispersion of molten sorbitol, with the aid of the piping 28 and the vibration mechanism 19, an equal amount of crystallised sorbitol in powder form is brought in, the mixture of globules and particles of powder taken place on the surface of the mass M.

The tank 1 turns at a speed of 7 revolutions/minute, has a diameter of 3.60 m and a depth of 1.20 m its inclination being 30°, and this permits the obtaining of granules with a diameter of 4 mm. We should mention that the average length of time that a given particle or globule which has been brought by the aforementioned means stays in the tank up till the time when, incorporated into a granule, it leaves tank 1, is in the order of half an hour. The granules, the size of which is about 4 mm, are kept at 90°C after being taken from the outlet of the tank 1 and routed into the rotating cylinder 4 in which they remain at this temperature for 5 hours.

At the outlet of the grinding plant 5, the sorbitol is sifted, the part recycled to the tank 1 being that with a particle size of less than 0.5 mm; the remainder appears in the form of a powder containing approximately 0.02% residual humidity and having a melting temperature of 95 to 98°C.

* * *

It should be mentioned that it is possible to manufacture by using the process and plant according to the invention, sweetened, coloured and/or flavoured powdered sorbitol by adding to the molten mass, for instance when it is inside vessel 3, sweetening agents such as, for instance, cyclamate or saccharine, colouring agents of the type used in the food industry and/or flavourings such as, for example, extracts of lemon, vanilla and acidulating agents such as, for example, citric acid.

EXAMPLE 2

By proceeding as in Example 1 and with

the purpose of manufacturing sweetened sorbitol, the molten mas, with a content in dry matter of 99.8% is brought into the vessel, storage tank or buffer 3, where same 1.33% of cyclamate of calcium is added.

The mixture thus obtained is homogenised by means of the stirrer 15. It is then continuously taken off as in Example 1, then dispersed on a bed of crystallised sorbitol already containing an equal amount of cyclamate, added during a preceding operation.

The mixture of the globules and particles of powder, containing the same amount of cyclamate is carried out on the surface of the mass M.

The length of stay inside the tank or dredger 1 is of the order of half an hour, and the speed of rotation of the latter is 7 revolutions per minute.

The granules then follow the same circuit as in Example 1 and go through the rotating cylinder 4, then into the grinding and sifting plant.

The residual humidity at the outlet is less than 0.05%.

EXAMPLE 3

As in Example 2 and with the purpose of manufacturing a sweetened sorbitol, the molten mass, with a content in dry matter of 99.8%, is brought into the storage tank or buffer 3, where 0.1% of saccharine is added; it is then taken off and dispersed on some molten sorbitol containing the same amount of saccharine.

The granules obtained at the outlet of the dredger 1 are routed into the rotating cylinder 4 and into the grinding and sifting plant.

EXAMPLE 4

With the aim of manufacturing a sorbitol which has the organoleptic characteristics of lemon, the molten mass, with a content in dry buffer of 99.8% is routed into the storage tank or buffer 3, where 2% of citric acid and 0.1% of E 104 Quinoline Yellow or E 102 Tartrazine Yellow, is added.

The molten mass, thus acidulated and coloured, is homogenised by means of the stirrer 15, then continuously taken off by the pump 16, and as in the preceding examples, dispersed on an equal amount of crystallised sorbitol, acidulated and coloured, as a result of a previous operation; the composition of the globules and particles of powder being the same and the same mixture being made on the surface of the mass M.

The granules which have the same size as that given in the preceding Examples follow the same circuit through the rotating cylinder and the grinding and sifting plant.

EXAMPLE 5

With the aim of manufacturing a sorbitol which has the organoleptic characteristisc of the cherry, the molten mass, with a content in dry matter of 99.8% is brought into the storage tank or buffer 3 where 0.001% by weight of E 127 Erythrosin, cherry flavouring and 1% of citric acid is added.

The mass is homogenised, then continuously taken off and pulverised on the surface of the mass M.

Consequently and whatever the method of execution adopted, we thus have provided a process for the manufacture of crystalline sorbitol which offers numerous advantages including in particular:

— that of being continuous

— that of ensuring a good yield with a stable quality

— that of being capable of automatic control.

We are aware of the Artificial Sweeteners in Food Regulations 1969 No. 1817, and insofar as our invention relates to the use of cyclamates, we make no claim to the use of the invention in contravention of the law. Subject to this disclaimer,

WHAT WE CLAIM IS:—

1. A process for the manufacture of crystalline particles of sorbitol, which comprises continuously mixing molten sorbitol having a dry matter content of at least 90% by weight with from 20 to 80% by weight of powdered sorbitol based on the total weight of molten and powdered sorbitol, the mixing being effected by simultaneously dispersing the molten and powdered sorbitol into an open rotating receptacle containing granules of conglomerated molten sorbitol and sorbitol powder whereby the molten and powdered sorbitol are mixed at the surface of the sorbitol contained in the receptacle, collecting sorbitol granules from the receptacle and crystallising molten sorbitol contained in said granules, the sorbitol in the receptacle being maintained in motion by the rotation of the receptacle and being maintained at a temperature of at least 90°C.

2. A process as claimed in Claim 1 in which the molten sorbitol is introduced into the receptacle in a sub-divided form.

3. A process as claimed in Claim 2 in which the molten sorbitol is introduced in the form of drops.

4. A process as claimed in Claim 2 in which the molten sorbitol is introduced in the form of jets.

5. A process as claimed in Claim 2 in which the molten sorbitol is introduced in the form of sheets or layers of sheets.

6. A process as claimed in any preceding claim in which the axis of rotation of the receptacle is inclined to the horizontal.

7. A process as claimed in any preceding claim in which the sorbitol granules are collected by overflow at the outlet of the receptacle.

8. A process according to any preceding claim in which at least particles of a diameter from 1 to 50 mm are collected.

9. A process according to any preceding claim in which molten sorbitol having a dry matter content of more than 98 weight % is brought to a temperature greater than 95°C and continuously mixed with a substantially equal amount of powdered sorbitol having a particle size of less than 5 mm, the dimensions, the orientation of the axis of rotation and the speed of rotation of the receptacle being selected so that the product collected from the receptacle appears in the form of granules of a diameter of from 1 to 50 mm.

10. A process according to Claim 9 in which the molten sorbitol is brought to a temperature of about 100°C.

11. A process according to any preceding claim in which the receptacle is in the form of an open tank or drum having a substantially flat bottom.

12. A process according to Claim 11 in which the axis of rotation of the receptacle makes an angle of from 25 to 45° to the horizontal.

13. A process according to any one of Claims 1 to 10 in which the receptacle is in the form of a sphere from which a dome has been removed.

14. A process according to any preceding claim in which the collected sorbitol granules are matured in order to increase their crystallinity by maintaining the granules at a temperature of from 50 to 95°C for from 1 to 20 hours while keeping the granules moving in a current of air.

15. A process according to Claim 14 in which the granules are maintained at a temperature of from 50 to 95°C for from 4 to 6 hours.

16. A process according to Claim 14 or Claim 15 in which the granules are maintained at a temperature of from 80 to 90°C.

17. A process according to any preceding claim in which sweetening, colouring, flavouring or acidulating agents are added to the molten sorbitol.

18. A process according to Claim 17 in which saccharine, lemon or vanilla extract and/or citric acid are added to the molten sorbitol.

19. A process for producing granular particles of sorbitol according to Claim 1 and substantially as hereinbefore described and exemplified.

20. Granular sorbitol whenever produced by a process according to any preceding claim.

MATHYS & SQUIRE,
Chartered Patent Agents,
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London, E.C.4.
Agents for the Applicants.

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1481846 COMPLETE SPECIFICATION

2 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 1

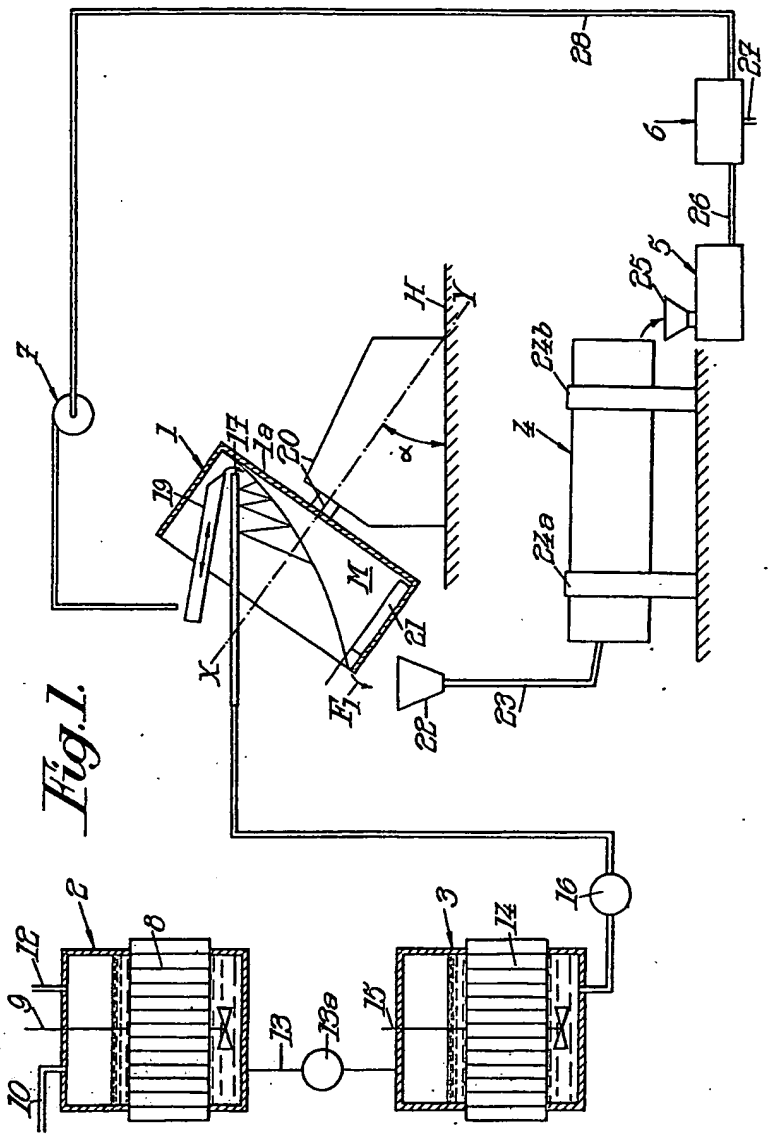

Fig.1.

1481846 COMPLETE SPECIFICATION
2 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheet 2

Fig. 2.

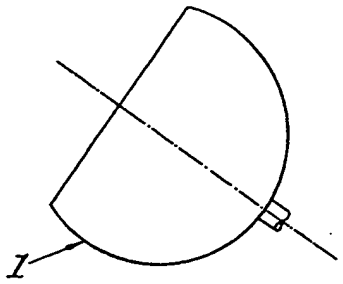

Fig. 3.